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HIGH TEMPERATURE CORROSION PREVENTIVE ADDITIVES FOR FLUOROCARBON POLYETHER FLUIDS

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TECHNICAL REPORT AFML-TR-67-210

SEPTEMBER 1967

AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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ROLAND E. DOLLE

FOREWORD

This report was prepared by the Fluid and Lubricant Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Directorate of Laboratories. Work was initiated under Project No. 7343, "Aerospace Lubricants", Task No. 734303, "Fluid Lubricant Materials", with Roland E. Dolle acting as project engineer. The report was submitted in May 1967. It covers work accomplished from 1 October 1964 to 15 March 1967.

The additive compounds used in the investigation resulted from in-house research efforts of the Polymer Branch (MANP), Nonmetallic Materials Division, Air Force Materials Laboratory. The author wishes to acknowledge the important contribution of MANP in synthesizing the additive materials. The author also expresses his appreciation to Frank J. Harsacky of the Fluid and Lubricants Materials Branch for his helpful suggestions and assistance.

This technical report has been reviewed and is approved.

R. L. ADAMCZAK
Chief, Fluid and Lubricant Materials Branch
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ABSTRACT

A new family of fluorocarbon polyether fluids was investigated. The results show that these fluids have potential for advanced lubricant and hydraulic fluid applications. The fluid class is thermally stable at 700°F, has good lubricating characteristics, and is nonflammable. A shortcoming of these fluids is their corrosive effect on ferrous and titanium alloys in oxidizing atmospheres above 500°F. A preliminary study was made by the Air Force Materials Laboratory (AFML) to determine the effectiveness of four perfluoroarylphosphine type of compounds as corrosion preventive additives in the fluorocarbon polyethers. Results showed the compounds, tris(pentafluorophenyl)phosphine, tris(pentafluorophenyl)phosphine oxide, tris(4-heptafluorotolyl)phosphine, and tris[4-(pentafluorophenoxy)tetrafluorophenyl]phosphine to be highly effective in reducing or completely eliminating the degradation of the fluid and the corrosion of ferrous and titanium alloys in oxidative environments to 650°F. Work is continuing to develop similar phosphine compounds having improved low volatility and better low temperature solubility in the fluorocarbon polyethers, because, although current work has proved the feasibility of the phosphine additives, further development is required for reduction to practice.

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SECTION I

INTRODUCTION

With the design and development of advanced high performance aerospace systems new fluids and lubricants are needed which can withstand severe thermal and oxidative stress at temperatures greater than 500°F without appreciable degradation. High temperature thermal and oxidative stability must be accompanied by good low temperature flow characteristics and satisfactory lubricating properties. State-of-the-art materials such as the super-refined mineral oils, diesters, and the polyphenylethers lack at least one of the important properties required for use in a high temperature lubricant system which is open to the atmosphere. For example the super-refined mineral oils have good low temperature flow properties, excellent lubricating ability, and are thermally stable at 700°F (Reference 1), but they do not have good oxidation resistance at high temperatures (References 2 and 3). The diesters, which meet current gas turbine engine oil requirements, cannot be expected to survive oxidatively at the 500°F level. The polyphenylether base fluids have shown good oxidation resistance at 550°F (References 4, 5, and 6) and are thermally stable at 850°F (Reference 6), but these materials have high pour points and relatively poor lubricating ability (References 5 and 7). In the current search for advanced fluids, one important target has been to obtain a gas turbine engine lubricant which is comparable to the polyphenylethers in oxidation and thermal stability, but one which also has better low temperature flow properties and better lubricating characteristics.

In 1962 a cooperative program was undertaken by the Air Force Materials Laboratory and E. I. duPont de Nemours and Company to fully develop a new class of fluorocarbon polyether high temperatre fluids. The fluid class was evaluated in laboratory experiments and performance tests to determine its potential for advanced lubricant and energy transfer applications (References 8 and 9). The bulk of the studies were made with the base fluids and one major shortcoming became apparent. At oxidizing temperatures above 500°F the fluids attacked ferrous and titanium alloys and in oxidation studies with titanium alloys significant bulk fluid deterioration occurred. However, certain aluminum alloys were not attacked by these fluids at 600°F and some superalloys containing nickel or cobalt were compatible with the fluids in oxidizing atmosphere to 700°F.

For future systems one could select only the superalloys during the design stage and eliminate most of the corrosion problem. This approach would be highly restrictive and could exclude the use of the fluorocarbon polyethers in advanced systems of the immediate future. An alternate route for obtaining a compatible fluid-metals system was believed to be through the use of additives. Successful in-house studies were made at AFML showing the effectiveness of various perfluorinated additive compounds in minimizing the degradation of the fluorocarbon polyethers and the corrosion of ferrous and titanium alloys in oxidative environments to 650°F. The results of these formulation studies are discussed herein following a brief review of some of the physical properties and lubricating characteristics of a typical gas turbine oil grade fluorocarbon polyether base fluid.

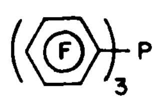
SECTION II

DISCUSSION

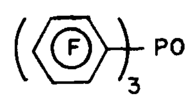
1. GENERAL

The fluorocarbon polyethers investigated are manufactured and sold by E. I. duPont de Nemours and Company and are supplied through duPont's Petroleum Chemicals Division under the trade name "Krytox" 143 (formerly known as PR-143). At present the chemical structure of the fluid class is proprietary to duPont. The various fluids currently available are chemically the same, but differ primarily in physical properties, such as viscosity and volatility, depending on the molecular weight distribution of the polymeric fractions comprising the fluids. In this study the lubricant formulations were prepared using as the base fluid a gas turbine engine oil grade fluorocarbon polyether.* The discussions to follow will refer to this type of fluid as simply GTO-143.

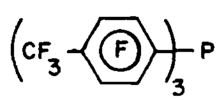
The compounds found to be effective additive systems in GTO-143 were from the class of perfluoroarylphosphines (References 10 and 11) consisting of:



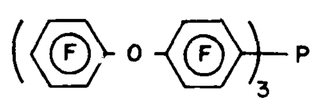
tris(pentafluorophenyl)phosphine
(FPP)



tris(pentafluorophenyl)phosphine oxide
(FPPO)



tris(4-heptafluorotolyl)phosphine
(FTP)



tris [4-(pentafluorophenoxy)tetrafluorophenyl] phosphine
(FPFPP)

An oxidation-corrosion (O-C) test similar to that described previously (Reference 12) was used for evaluating the compatibility of GTO-143 base fluid and the new perfluoroarylphosphine formulations with various metals in an air environment. One liter of dry air per hour was bubbled through a 20 milliliter fluid sample via an air entry tube for a period of 24 hours. Various small metal washers were mounted on the air entry tube which was immersed in the

*duPont's designation for the current gas turbine engine oil grade is "Krytox" 143 AC.

fluid during the test. The O-C apparatus is shown in Figures 1 and 2. An overboard test (no reflux condenser) was used and an aluminum block bath was the heat medium (Reference 13). The criteria for determining the extent of bulk fluid oxidation were fluid evaporation loss, percent change in kinematic viscosity at 100°F (Reference 14) using a semimicro viscometer, and change in acid number (mgKOH/g) using a potentiometric titrator (Reference 15). The effect of the O-C test on the metal specimens was defined by the change in weight (mg/cm^2) of each metal after the test.

Most of the tests employed for determining the general physical properties of GTO-143 base fluid were made according to Federal test methods (Reference 16) and standards set forth by the American Society for Testing and Materials. The lubricating properties described were obtained from four-ball wear tests using a Shell Four-Ball Wear Tester (Reference 17).

2. PHYSICAL PROPERTIES AND FOUR-BALL WEAR CHARACTERISTICS OF A GTO GRADE OF FLUOROCARBON POLYETHER BASE FLUID

Some physical property data for a typical GTO-143 base fluid are summarized in Table I. The operational viscosity-temperature range for the fluid is in the neighborhood of slightly above 0° to 700°F. Actually the viscosity-temperature range and volatility characteristics are varied for different grades of fluid depending on the molecular weight distribution of the polymeric fractions comprising them (Reference 9).

GTO-143 is nonflammable. No autogenous ignition, flash, or fire points were found up to 1000°F. Futhermore, the fluid does not flash or burn after contact with hot manifold temperatures to 1240°F (Reference 8).

GTO-143 has good thermal stability at 700°F, and adequate specific heat and thermal conductivity for most high temperature applications. The bulk modulus of the fluid is considered fair. Because of the chemical nature of the fluorocarbon fluid its density is about 1.9 gm/cc at room temperature.

The GTO-143 base fluid demonstrated good lubricating characteristics in four-ball wear studies under a variety of conditions (Table II). The wear scar values were generally less than 0.5 millimeter for loads up to 10 kilograms at 400°F. At a 40 kilogram load the wear scars measured one millimeter or less. The appearance of the fluid remained unchanged in all experiments.

3. COMPATIBILITY OF PERFLUOROARYLPHOSPHINE ADDITIVE FORMULATIONS WITH FERROUS ALLOYS

Formulations resulting from the addition of a perfluoroarylphosphine additive to GTO-143 were much more compatible with ferrous alloys than uninhibited GTO-143, as shown in Table III. After 48 hours at 600°F considerable metal corrosion occurred in the base fluid; whereas, corrosion was eliminated or reduced to nil in the formulations which contained 0.10 wt. % FPFPP. The 4140 steel and M-2 and M-50 tool steel specimens had weight changes over 5 mg/cm^2 in the base fluid, but in the formulation the weight changes for these metals were only about 0.1 mg/cm^2 or less.

Similar benefits were obtained in 24 hour tests at 650°F with GTO-143 formulations containing 0.10 and 0.20 wt. % FPFPP. As expected, corrosion was significantly greater in the uninhibited fluid at 650°F than it was at 600°F, yet in most cases the weight changes of the metals immersed in the formulations at 650°F were reduced to the same low level as the weight changes of the metals in the formulations at 600°F. The notable exceptions were the

M-2 and M-50 tool steel specimens. Although the weight changes for these metals were substantially reduced at 650°F in the formulations, significant corrosion was observed.

Metal corrosion in the base fluid was not as pronounced after 24 hours at 600°F as in the 48 hour tests at 600°F or the 650°F tests; nevertheless, the performance of all the perfluoroarylphosphine-containing formulations was still quite dramatic. Included in these 600°F tests were GTO-143 formulations which contained FPP, FPPO, or FTP. These additives were just as effective in reducing corrosion as FPFPP at 600°F, but because of their higher volatility they were not used at 650°F. The most effective of these lower molecular weight additive compounds was FTP, of which concentrations as low as 0.10 wt. % were effective; the optimum concentration was about 0.25 wt. %.

In the O-C experiments with the ferrous alloys neither the inhibited nor the uninhibited fluid had significant bulk property change; the viscosity changes were nil and no acid numbers were found after any of the tests. Slightly more fluid loss was observed for the uninhibited fluid, especially during the 650°F tests. Those ferrous alloy specimens immersed in the formulations which had no more than 0.1 mg/cm^2 weight change (considered nil) were only slightly discolored (usually a light gold with some violet). The specimens with 0.1 to 0.2 mg/cm^2 change were also only slightly discolored, but had some gray smears, which were usually observed around the imperfections on the metal surface. (It should be noted that the O-C stability requirement of most high temperature lubricant specifications limit metal specimen weight change to 0.2 mg/cm^2.) The metals which had up to 0.35 mg/cm^2 change (either in the base fluid or the formulations) had gray smears that were more pronounced, but the smears usually did not cover more than about one-fourth the total surface area of the metal. The metals with changes near one mg/cm^2 or greater were corroded badly resulting in a rough dark red-brown and sometimes black appearance over the entire surface area.

Most of the ferrous metals that were immersed in the uninhibited fluid for 48 hours at 600°F and 24 hours at 650°F had substantial weight gains, but some specimens lost weight. The weight increases were believed to be due to the accumulation of oxide deposits on the metal surface; an erroding effect occurred on the metals which lost weight. The 52100 bearing steel and M-2 and M-50 tool steels gained weight and the 410 and 440-C stainless steel specimens lost weight. 4140 steel lost weight after 48 hours at 600°F but gained weight at 650°F. Considerable flaking of the 4140 specimen during the 600°F tests was observed which would explain the weight loss.

4. COMPATIBILITY OF PERFLUOROARYLPHOSPHINE ADDITIVE FORMULATIONS WITH TITANIUM AND TITANIUM ALLOYS

The O-C performance of the GTO-143 base fluid with titanium metals (Table IV) was different from that of the ferrous metals. When the uninhibited fluid was exposed to oxidative stress in the presence of titanium and titanium alloys, not only were the metals attacked, but the fluid itself was severely deteriorated, especially at 650°F. However, with the addition of as little as 0.01 wt. % FPFPP, metal corrosion and fluid degradation were noticeably reduced at 600°F and fluid deterioration and metal corrosion were completely eliminated with 0.05 wt. % FPFPP. Concentrations of FTP, FPP, and FPPO ranging from 0.25 to 0.50 wt. % were also effective in eliminating fluid degradation and corrosion of the titanium metals at 600°F.

At 650°F uninhibited GTO-143 deteriorated very badly resulting in 50% fluid loss and 91.8 % viscosity decrease. The pure titanium and aluminum-manganese-titanium alloy were severely attacked. In sharp contrast, the formulations containing 0.10 wt. % and 0.20 wt. % FPFPP did not degrade and metal corrosion was nil. It should be noted that in the 650°F test

with the uninhibited fluid, the aluminum-vanadium-titanium alloy did not have a significant weight change, but the surface of the metal was blackened. It is not uncommon for some of the titanium alloys themselves to have negligible weight changes even though the GTO-143 in which they are immersed degrades badly. The titanium specimens with a large weight increase also had a smooth blackened surface. Those titanium metals which were immersed in the formulations normally were light gold in color or sometimes dark blue.

5. VOLATILITY AND SOLUBILITY OF FORMULATED PERFLUOROARYLPHOSPHINE ADDITIVES

In the O-C tests at 600°F an appreciable collection of FTP, FPP, and FPPO was observed near the top of the O-C tube long before the 24 hour tests were completed. In comparison very little FPFPP was observed after 24 hours or even 48 hours at 600°F. The fact that the FTP, FPP, and FPPO are more volatile than FPFPP is understandable when the molecular weight of the compounds is considered. However, variation in degree of volatility among the three lower molecular weight additives was observed. FPP and FPPO collected near the top of the O-C tube in appreciable amounts in 600°F tests after only 2 to 3 hours; whereas, FTP did not accumulate as rapidly. Additive volatility at the test temperature is closely associated with the additive concentration needed for optimum protection. The less volatile FPFPP was effective in 600°F tests in concentrations as low as 0.01 wt. %, with 0.05 to 0.10 wt. % giving the most satisfactory results. To achieve optimum benefit using FTP, about 0.25 wt. % of the additive was required and from 0.25 to 0.50 wt. % FPP and FPPO was needed.

Only the formulations containing the less volatile FPFPP were evaluated in 48 hour tests at 600°F and in 650°F tests for 24 hours. After the 650°F tests some of the FPFPP additive had collected near the top of the O-C tube. At 650°F, concentrations of FPFPP of 0.20 wt. % generally gave better results than 0.10 wt. % of the same additive. According to strictly empirical observations during 600° and 650°F O-C tests, it appears that the volatility of the additives investigated decreases in the following order:

$$\mathrm{FPP} > \mathrm{FPPO} > \mathrm{FTP} > \mathrm{FPFPP}$$

The solubility of the perfluoroaryl type of additive compounds in GTO-143 was also measured rather empirically by dissolving various concentrations of the additive in the fluid with heating and stirring, and allowing the hot formulation to cool slowly to room temperature. During cooling, the formulations were observed for additive precipitation. Formulations in which no additive precipitation occurred after several days at room temperature were cooled to lower temperatures. These additive solubility approximations revealed the following order of decreasing solubility in GTO-143:

$$\mathrm{FTP} > \mathrm{FPP} > \mathrm{FPPO} > \mathrm{FPFPP}$$

FTP remains soluble in the fluid in concentrations of 0.25 wt. % for several days below room temperature, but eventually some precipitation occurs. Identical concentrations of FPP and FPPO begin precipitating within several hours at low temperatures. FPFPP is soluble in the fluid for several days at room temperature only in concentrations below 0.02 wt. %.

The most soluble additive was FTP and this was believed due to the solublizing effect of the perfluoromethyl group. It is believed that if the substituents attached to the phenyl radical were perfluoroethyl or perfluoropropyl, even better low temperature solubility would be obtained. Nevertheless, the additives investigated could still be useful in certain specialized applications where the formulation could be held to 50° to 75°F above room temperature. Certainly the formulations containing even the least soluble additives in high concentration would be useful as "base fluids" for greases where low temperature solubility is unimportant if solubility and activation are achieved at the higher operating temperatures.

SECTION III

CONCLUSIONS

The fluorocarbon polyether high temperature fluids are an extremely interesting new class of materials and have potential for advanced lubricant application. The fluids have good thermal stability, good lubricating characteristics, and are nonflammable. Although the fluids are known to have good oxidative stability to 700°F in the absence of metals, a serious shortcoming is their corrosive effect on ferrous and titanium alloys above 500°F. Evaluation of formulations composed of a gas turbine oil grade fluorocarbon polyether and various perfluoroarylphosphine types of additives resulted in the elimination or a significant reduction of metal corrosion and fluid degradation in air to temperatures of 650°F. Specifically, in concentrations ranging from 0.05 to 0.50 wt. %, the compounds FPP, FPPO, FTP, and FPFPP were highly effective as anticorrosion additives in the fluid. The additive concentrations used depended primarily on the volatility of the additive rather than on its inherent effectiveness. The incorporation of a perfluoromethyl group on the perfluorophenyl radicals in FPP significantly improved the solubility of the compound.

The inhibitive mechanism of the new additives was not investigated. However, it may be assumed that the additives function in such a manner as to inhibit incipient degradation of the fluorocarbon polyether, since the products of such degradation can cause corrosion. In turn, the corrosion products themselves, which are believed to be mostly metal oxides and metal fluorides, can catalyze further fluid decomposition, as in the case when titanium is present. A second way in which the additives may inhibit corrosion is through the formation of a thin film or coating to protect the metal from attack, thus preventing production of the harmful metal oxides and fluorides.

Although the solubility of the new additives in the fluorocarbon fluid was not the most desirable, analogs having better solubility can be made. Nevertheless, fluid formulations containing additives with low solubility could be utilized in grease formulations or in other specialized applications. Continued development work is required and is underway to develop similar phosphine compounds having improved low volatility and better low temperature solubility in the fluorocarbon polyethers.

TABLE I

PHYSICAL PROPERTIES OF A TYPICAL GTO-143 BASE FLUID

Viscosity, centistokes @ 0°F	42,000[a]
100°F	283
210°F	25.5
400°F	3.64
500°F	1.90
550°F	1.45
700°F	0.8
Viscosity index	115
Pourpoint, °F	-25
Autogenous ignition temperature, °F	>1,000
Flash point, °F	> 940[b]
Fire point, °F	> 940[b]
Evaporation[c], 6-1/2 hrs, % wt. loss @ 550°F	11.9
600°F	34.8
Volatility by TGA[d], % wt. loss @ 464°F	0
680°F	10
739°F	25
792°F	50
829°F	75
889°F	100
Thermal stability[e], 6 hrs @ 700°F	
viscosity change @ 100°F, %	-3.3
neut. no. increase, mg KOH/g	0.0
metal wt. change, mg/cm^2	
M-10 tool steel	0.00
52100 bearing steel	+0.02
naval bronze	+0.06[f]
Specific heat, BTU/lb/°F @ 100°F	0.241
200°F	0.260
300°F	0.279
400°F	0.297
500°F	0.316
Thermal conductivity, BTU/hr $(ft)^2$ (°F/ft) x 10^3 @ 100°F	52.1
200°F	52.0
300°F	51.8
400°F	51.6
500°F	51.5
Bulk modulus, isothermal secant 3000 psi, psi x 10^{-3} @ 100°F	142
425°F	48
Density, gm/cc @ 0°F	1.9567
100°F	1.8838
200°F	1.7865
400°F	1.5963
500°F	1.5025

a. Hydraulic fluid grade has a 0°F viscosity of 3,600 cs and other gas turbine oil grades have 0°F viscosities as low as 22,000 cs.
b. The sample had completely evaporated at 940°F.
c. As per ASTM-D972.
d. Determinations made in platinum crucible at atmospheric pressure with 0.25 ft^3/hr helium sweep and heating rate of 6°C/min.
e. Penn State thermal stability test at constant temperature as per MIL-H-27601 (Reference 18).
f. Initially +0.24. Upon wiping, the bronze catalyst weight change dropped to +0.06.

TABLE II

FOUR-BALL WEAR CHARACTERISTICS OF A TYPICAL GTO-143 BASE FLUID

Shell Four-Ball Wear Test Conditions		Average wear scar diameter, millimeters.
600 RPM, 167°F, 2 hrs, 52100 balls @	1 kg load	0.153
	10 kg load	0.453
	40 kg load	0.643
600 RPM, 400°F, 2 hrs, 52100 balls @	1 kg load	0.168
	10 kg load	0.304
	40 kg load	0.752
600 RPM, 400°F, 2 hrs, M-10 balls @	1 kg load	0.176
	10 kg load	0.230
	40 kg load	0.680
1200 RPM, 167°F, 2 hrs, 52100 balls @	1 kg load	0.308
	10 kg load	0.452
	40 kg load	0.782
1200 RPM, 400°F, 2 hrs, 52100 balls @	1 kg load	0.197
	10 kg load	0.520
	40 kg load	1.050
1200 RPM, 400°F, 2 hrs, M-10 balls @	1 kg load	0.218
	10 kg load	0.344
	40 kg load	0.719

TABLE III

COMPATIBILITY OF GTO-143 FORMULATIONS WITH VARIOUS FERROUS ALLOYS IN AIR

Additive	Test temp., °F	Test time, hrs	Fluid loss, %	Kinematic viscosity change @ 100°F, %	Acid number increase, mg KOH/g	Corrosion Effect[a]					
						4140	52100	M-2	M-50	410SS	440C
None	600	24	1.9	+1.8	0	+0.73	+0.24	+1.07	+0.66	+0.23	+0.35
0.05 wt. % FPFPP	600	24	0.3	+1.6	0	+0.10	+0.06	+0.13	+0.08	+0.05	+0.02
0.20 wt. % FPFPP	600	24	0.3	+3.9	0	+0.02	+0.07	+0.30	+0.08	+0.05	+0.04
0.10 wt. % FTP	600	24	1.0	+2.5	0	+0.15	+0.11	+0.11	+0.04	-0.01	0.00
0.25 wt. % FTP	600	24	0.3	+1.4	0	+0.10	+0.09	0.00	+0.01	+0.01	0.00
0.50 wt. % FTP	600	24	0.5	+1.4	0	+0.09	+0.05	+0.21	+0.10	+0.01	+0.03
0.25 wt. % FPP	600	24	1.3	+2.5	0	+0.31	+0.11	+0.24	+0.10	0.00	0.00
0.50 wt. % FPP	600	24	0.7	+2.1	0	+0.04	0.00	+0.11	+0.08	-0.01	+0.07
0.40 wt. % FPPO	600	24	0.8	+1.4	0	+0.06	+0.04	+0.20	+0.13	0.00	+0.01
None	600	48	3.6	+1.8	0	-5.85[b]	+1.66	+6.91	+5.18	-0.55	-1.29
0.10 wt. % FPFPP	600	48	0.3	+2.4	0	+0.08	+0.07	+0.11	+0.07	+0.03	0.00
0.20 wt. % FPFPP	600	48	0.5	+2.7	0	+0.06	+0.17	+0.32	+0.33	+0.03	-0.01
None	650	24	6.5	+1.2	0	+6.38	+2.59	+6.98	+5.67	-2.46	-4.52
0.10 wt. % FPFPP	650	24	2.0	+2.1	0	+0.11	+0.18	+2.04	+0.21	-0.19	-0.03
0.20 wt. % FPFPP	650	24	0.3	+1.8	0	+0.04	+0.06	+1.35	+0.55	+0.09	+0.08

a. Weight change of metal specimens in milligrams per square centimeter.
b. Much flaking occurred.

TABLE IV

COMPATIBILITY OF GTO-143 FORMULATIONS WITH TITANIUM AND TITANIUM ALLOYS IN AIR

Additive	Test temp., °F	Test time, hrs	Fluid loss, %	Kinematic viscosity change @ 100°F, %	Acid number increase, mg KOH/g	Corrosion Effect[a]		
						Pure titanium	4%Al-4%Mn alloy	6%Al-4%V alloy
None	600	24	7.8	- 7.4	0.1	-0.09	-0.32	+0.16
0.01 wt. % FPFPP	600	24	3.3	- 0.4	0	+0.06	+0.04	0.08
0.05 wt. % FPFPP	600	24	0.2	+ 1.4	0	+0.02	+0.02	0.00
0.10 wt. % FPFPP	600	24	0.3	+ 1.9	0	0.00	+0.01	0.00
0.25 wt. % FTP	600	24	0.3	+ 1.8	0	0.00	0.00	+0.01
0.50 wt. % FTP	600	24	0.3	+ 1.8	0	+0.02	+0.01	+0.02
0.50 wt. % FPP	600	24	0.5	+ 3.0	0	+0.01	0.00	+0.02
0.50 wt. % FPPO	600	24	0.8	+ 3.3	0	+0.06	+0.03	+0.02
None	650	24	50.0	-91.8	0.1	-0.60	-2.60	+0.01[b]
0.10 wt. % FPFPP	650	24	2.3	+ 1.4	0	+0.13	+0.07	+0.09[c]
0.20 wt. % FPFPP	650	24	0.2	+ 1.8	0	+0.08	+0.12	+0.06[d]

a. Weight change of metal specimens in milligrams per square centimeter.
b. Gray-black surface.
c. Blue-violet discoloration with some gray smears.
d. Blue-violet discoloration.

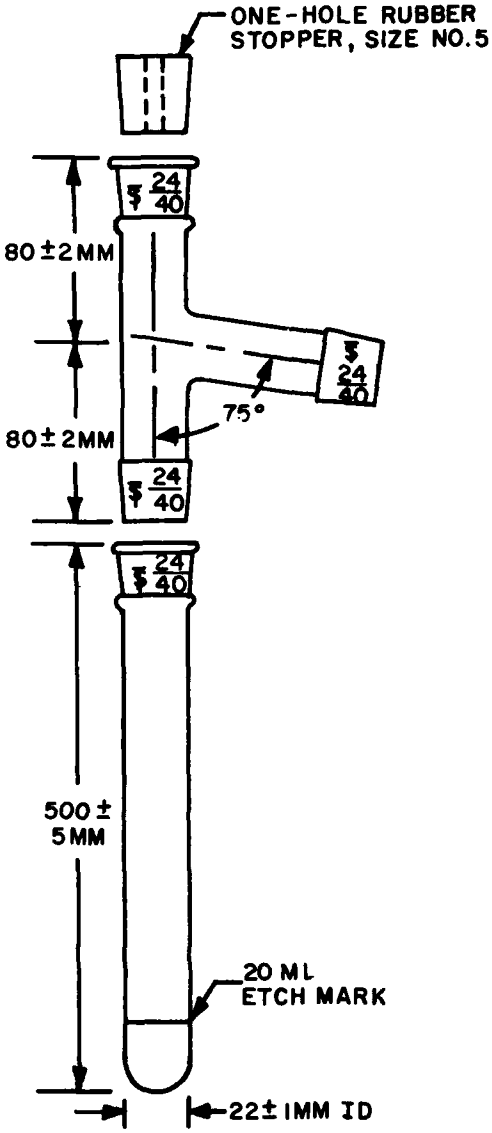


Figure 1. Oxidation Test Tube and Takeoff Adaptor

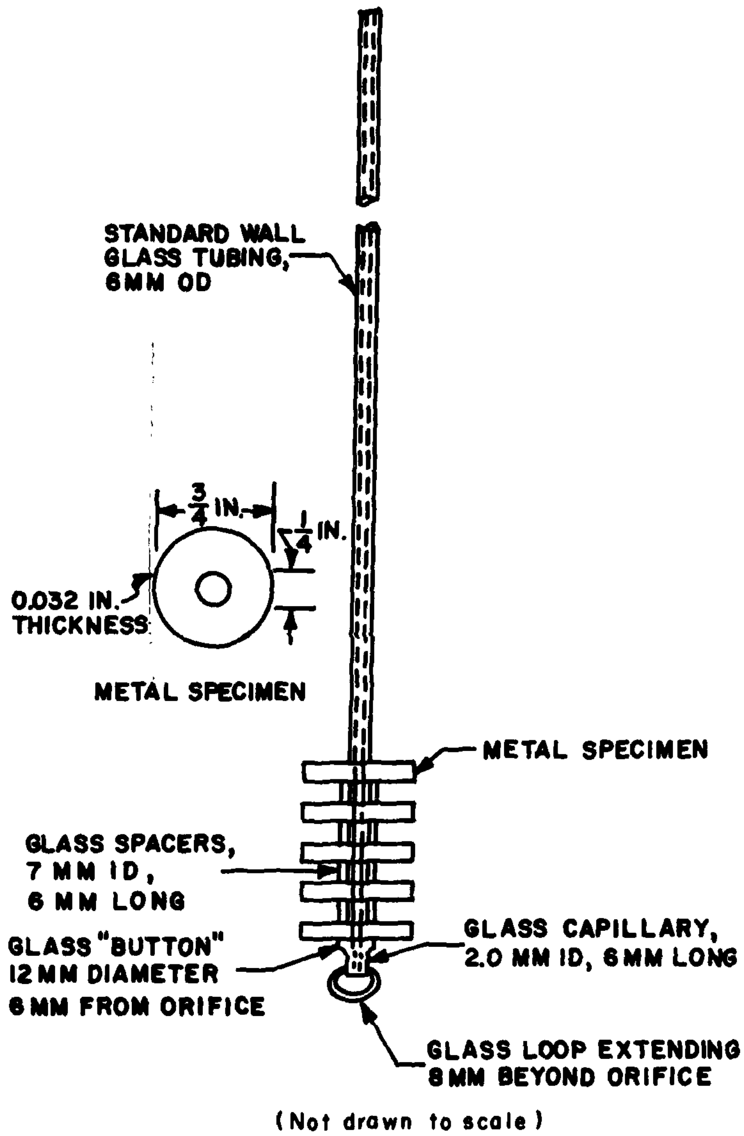


Figure 2. Air Entry Tube With Metal Specimens

SECTION IV

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13. ABSTRACT

A new family of fluorocarbon polyether fluids was investigated. The results show that these fluids have potential for advanced lubricant and hydraulic fluid applications. The fluid class is thermally stable at 700°F, has good lubricating characteristics, and is nonflammable. A shortcoming of these fluids is their corrosive effect on ferrous and titanium alloys in oxidizing atmospheres above 500°F. A preliminary study was made by the Air Force Materials Laboratory (AFML) to determine the effectiveness of four perfluoroarylphosphine type of compounds as corrosion preventive additives in the fluorocarbon polyethers. Results showed the compounds, tris(pentafluorophenyl)phosphine, tris(pentafluorophenyl)phosphine oxide, tris(4-heptafluorotolyl)phosphine, and tris[4-(pentafluorophenoxy)tetrafluorophenyl] phosphine to be highly effective in reducing or completely eliminating the degradation of the fluid and the corrosion of ferrous and titanium alloys in oxidative environments to 650°F. Work is continuing to develop similar phosphine compounds having improved low volatility and better low temperature solubility in the fluorocarbon polyethers, because, although current work has proved the feasibility of the phosphine additives, further development is required for reduction to practice.

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